NOVEL METHOD FOR OBSERVATION OF THE THERMAL PROPERTIES OF THIN FILM WITH MODULATED CO_2 LASER[1]

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Abstract

We have successfully performed the measurement of thermal diffusivity of thin metal film combining a fast infrared radiation thermometer with Mercury Cadmium Telluride (MCT) detector and the CO_2 laser modulated at an rf, from DC to 2MHz. The laser output beam is modulated by an AOM (Acousto-Optic Modulator) and is led to the front surface of the blackened copper thin film (10µm thick, 9.5mm in diameter). The thermal radiation from the back surface of the sample is detected. From observed phase delay in the detected signal of 0.68 radian to the input laser beam, the thermal diffusivity is obtained to be 1.11×10^{-4} m^2/s, which agrees well with the value of 0.99×10^{-4} m^2/s calculated from the data in the references. The method is generally applicable for measurements of thermal properties of nano/micro materials.

KEYWORDS: fast infrared radiation thermometer, CO_2 laser, periodic heating, MCT, thin film

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1. INTRODUCTION

Recent rapid progresses in semiconductor industry and material science require a measurement technique capable of detecting a fast temperature change in a very small area of the sample surface. A non-contact method is also important, so as to be free from contamination of the sample. The conventional method with a thermocouple or a resistance thermometer may not be applicable for this purpose. A high-speed thermo-reflectance method may be employed in the measurements [1]. However, the above method is applicable only for flat surfaces with specular reflection. The detection technique of thermal radiation from the sample specimen irradiated by a laser flash or a periodically modulated laser beam is complementarily used for diffusive surfaces [2-6].

So far the mid-infrared radiation thermometer with a laser flash excitation was employed [7]. The dynamic range, however, was limited from DC to 10kHz. In the present research, a radiation thermometer in 10μm range with a high-speed photovoltaic-type of Mercury Cadmium Telluride (MCT) detector has been developed. A wider dynamic range up to 2MHz has been achieved. A very small area is irradiated by the 10.6μm cw CO_2 laser modulated at the radio frequency. This method allows us to measure the thermal properties of small and thin specimens, which are transparent at the visible region and opaque at the infrared region.

2. EXPERIMENTAL APPARATUS AND PROCEDURE

The present radiation measurement system can detect the rapid temperature change in the radiating substance at micro-second region. The required specifications of the radiation thermometer which is to be applied for measurements of thermal diffusivity of thin films are as follows: the measurement temperature ranges from room temperature to over 500^{o}C, the dynamic range is from DC to 1MHz, the temperature resolution is less than 0.1^{o}C and the spatial resolution on the sample surface is less than 1mm. The actually developed radiation thermometer has the dynamic range of DC to 2MHz, temperature resolution of 50mK and spatial resolution of 500μm. The MCT detector cooled with liquid nitrogen has the peak sensitivity at 10μm. Parabolic mirror optics is employed as an imaging system of the thermometer. The focal lengths of the primary and secondary parabolic mirrors are 127mm and 254mm, respectively. The diameter of the detector aperture is 1mm. The radiation shield placed in front of the detector prevents the background radiation from entering the detector. The temperature of the radiation shield is kept at 77K. Detailed descriptions of design and construction of the radiation thermometer is presented in another paper [8]. The radiation thermometer is shown in Fig.1.

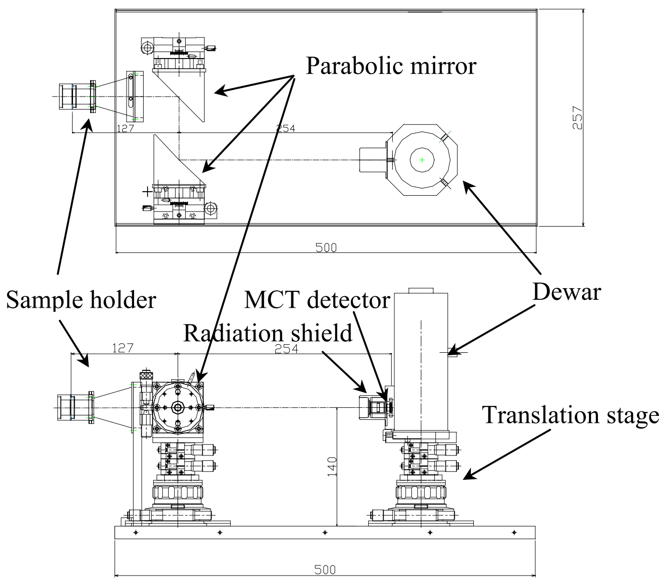

Fig.1.Top and side views of the radiation thermometer.
The sizes of components are shown in [mm].

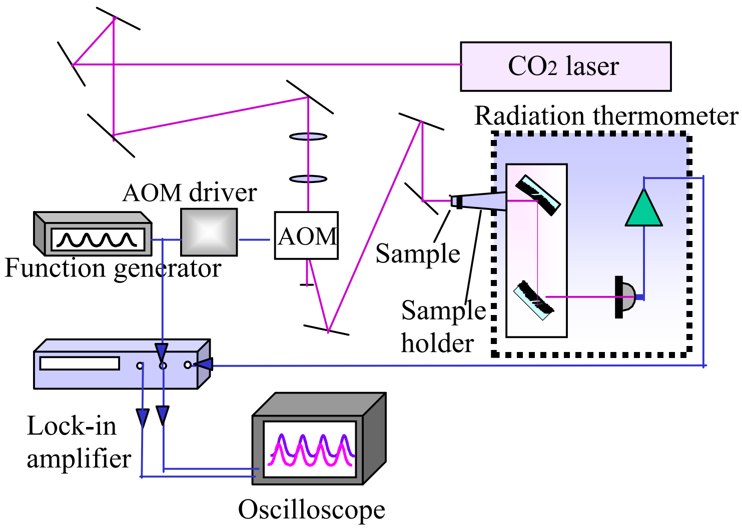

Fig.2. Schematic diagram of experimental apparatus

A block diagram of the periodic heating measurement system is shown in Fig.2. The system is composed of two parts, the pump part and the probe part. The power and the frequency of the 10.6 μm cw CO_2 laser are stabilized by a feed back system with a piezo electric transducer (PZT). The maximum output power of the laser is 4W. It is attenuated by using an aperture. The laser is modulated by the AOM (Acousto-Optic Modulator) at the frequency from 10 kHz up to 2MHz. The function generator provides the modulation signal to the AOM, the reference signal to the lock-in amplifier, and the reference time scale on the oscilloscope. The signal without sample gives the zero-delay signal on the CRT display. The modulated laser light irradiates the front surface of the sample mounted on a taper shaped sample holder. The sample holder is made of aluminum and treated with a low reflectance black coating. The radiation from the back surface of the sample is focused to the detector aperture of 1mm diameter by two parabolic mirrors. The modulated laser beam irradiates the area $1.77\times10^{-6}m^2$ of the sample.

The time response of infrared thermometer is evaluated by detecting this modulated laser beam. The output signal from the thermometer responds to the high frequency modulation from 1kHz to 2MHz. The result at 1MHz is shown in Fig.3. The time delays in the detection system and in the AOM are also evaluated to be negligible. Accordingly, the phase delay can be measured between the signals with and without the sample.

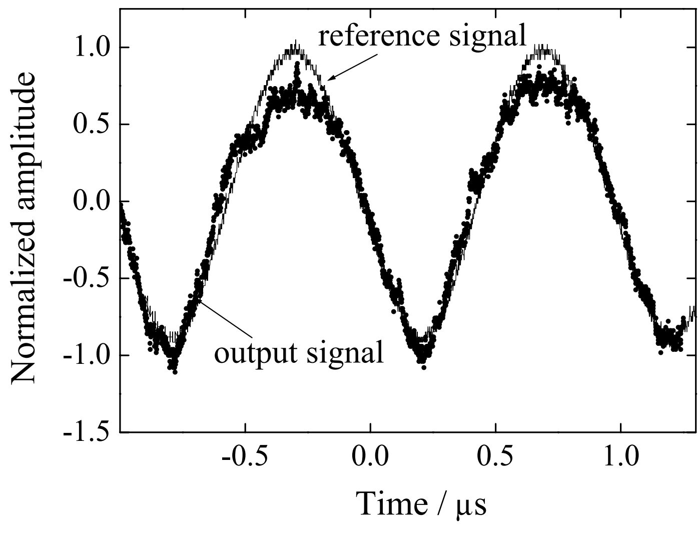

Fig.3. The output signal from the radiation thermometer and the reference signal showing good time response of the thermometer.

3. METHOD OF ANALYSIS

Since the thickness of the sample is much smaller than the diameter of the sample, the one-dimensional model of thermal conduction is employed (Fig.4).

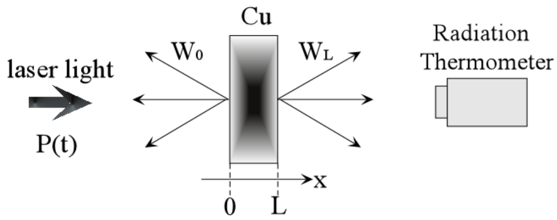

Fig.4. One-dimensional model of thermal conduction. The thin film is irradiated by the modulated laser beam, ($P(t)$), and becomes an equilibrium by emitting thermal radiation(W_0 ,W_L) from the front and back surfaces. The thermometer detects W_L.

The differential equation of thermal conduction is given by

$$\alpha\left(\frac{\partial^2 T(x,t)}{\partial x^2}\right)=\frac{\partial T(x,t)}{\partial t} \quad , \tag{1}$$

where $T\,(x,t)$ is the temperature as a function of the distance from the sample surface and the time t and α is the thermal diffusivity. Equation (1) satisfies the boundary conditions at the front and back surfaces,

$$P = W_0 + \left(k\frac{\partial T(x,t)}{\partial x}\right)_0 \tag{2}$$

$$\left(k\frac{\partial T}{\partial x}\right)_L = W_L \tag{3}$$

where P is the injected laser power, W_0 the radiation power from the front surface of the specimen, k the thermal conductivity, L the length from the front surface, and W_L the radiation power from the back surface of the specimen. $T\ (x,t)$ can be approximated by a sinusoidal function with a phase delay of δ_x to the phase of input laser light. At the back surface, x=L, the phase delay is given by [9],

$$\tan\delta_L = \frac{b(\tan B - \tanh B) + 2aB\tan B\tanh B + 2B^2(\tan B + \tanh B)}{b(\tan B + \tanh B) + 2aB - 2B^2(\tan B - \tanh B)} \tag{4}$$

where B,a and b are the non-dimensional parameters. These are given by,

$$\begin{aligned} B &= L(\omega/2\alpha)^{1/2} \\ a &= L(c_L + c_0) \approx 2Lc_L \\ b &= L^2 c_L c_0 \approx L^2 {c_L}^2 = a^2/4 \end{aligned} \tag{5}$$

where

$$c_x = \left(\frac{\partial W}{\partial T} \Big/ k \right)_x \tag{6}$$

The value of the parameters, a and b, are obtained to be a=7.4×10^{-5}, b=1.4×10^{-9} in the present experimental conditions, L=10μm and ω=2π×10^6 Hz. From the observed value of the phase delay δ_L the parameter B and then the thermal diffusivity α can be obtained.

4. EXPERIMENTAL RESULTS AND DISCUSSION

The system is applied to a copper thin film with the thickness of 10μm and the diameter of 9.5mm. Both surfaces of this specimen are blackened by the carbon and become highly emissive at 10.6 μm. The specimen is placed on a thermally insulated holder. Since the time constant of thermal diffusion of the present specimen is in the order of 1μs, the modulation frequency is set to 1MHz. We measured the modulated radiation signal from the back surface of the specimen and the modulated signal without specimen. By the least square fitting of the sinusoidal curve to the observed signal, we obtained the phase delay of 0.68 radian at 1MHz. The experimental results are shown in Fig.5. From Eq. (4) the thermal diffusivity α is obtained to be $\alpha = 1.11\times10^{-4}$ m^2/s.

We calculate the literature value [10] of $\alpha = k/(C\bullet\rho)$ from k : thermal conductivity, C ; thermal capacity, and ρ : density at the DC part of the temperature T. T is estimated by balancing the input laser power with the radiation power from both surfaces of the sample. The result is $\alpha = 0.99\times10^{-4}$ m^2/s which agrees well with the experimental value of α = 1.11×10^{-4} m^2/s.

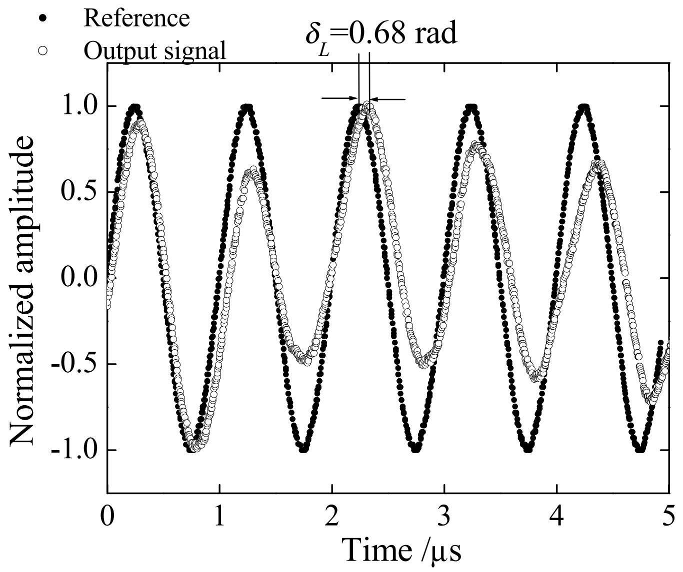

Fig.5. The sinusoidal signals of the thermal radiation and the laser beam. The open circles show the signal from the back surface of the copper metal film and the closed circles show the reference signal of input laser beam. The phase delay of 0.68 radian is observed.

5. Conclusion

We have developed the novel method for observation of the thermal properties using the fast radiation thermometer and the modulated CO_2 laser beam. The characteristic feature of this system is that the phase delay in Eq.(4) changes sensitively as a function of *B*, which is inversely proportional to the square root of the thermal diffusivity. Therefore, we can determine the thermal diffusivity with high accuracy. We need to accurately measure only the phase delay in the case of periodic heating method.

The important point of this system is that we are using the mid-infrared laser as an excitation source. Therefore, we can investigate materials such as glass, sapphire, and some semiconductors, which have absorption bands at 10 μm but are transparent at the visible region. The blackening, which may introduce uncertainty in the measurement, is not necessary if the specimen is opaque at the infrared region. We are currently working to measure transparent specimens.

To improve and extend the present experimental system, we are trying to reduce the sample temperature by attenuating the DC part of the laser radiation and by attaching a radiator to the sample holder.

We are also going to measure the thermal properties of the layer sample deposited on glass substrates. The substrates absorb infrared laser radiation and prevent the layer sample from being deteriorated. This measurement system can be applied for any sample deposited on the glass substrates.

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